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ning of each regular issue of the PCT Gazette.

(54) Title: MANUFACTURING METHOD OF SEMICHEMICAL MECHANICAL PULP FROM CORNSTALK

(57) Abstract: Provided is a manufacturing method of semichemical mechanical pulp from cornstalks capable of offering environment-friendly pulp by reducing use of chemicals, offering a method for manufacturing new Korean pulp from comstalks and enabling manufacture of characteristic paper. The method comprises the processes of crushing cornstalks into chips, applying heat and pressure to the crushed chips with such alkaline solution as caustic soda, refining them and bleaching them by adding such oxidizer as hydrogen peroxide.

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# **Description**

# MANUFACTURING METHOD OF SEMICHEMICAL MECHANICAL PULP FROM CORNSTALK

#### **Technical Field**

[1] The present invention relates to a manufacturing method of semichemical mechanical pulp from cornstalks. More particularly, it relates to a manufacturing method of semichemical mechanical pulp capable of manufacturing environment-friendly pulp from cornstalks and manufacturing characteristic paper.

[2]

[4]

[6]

[7]

# **Background Art**

[3] With the rise of national income, Korea has become the world's seventh largest paper (including publications, newspapers, publishing cardboards, kraft paper, bulk paper, etc.) consumer and at the same time world's ninth largest paper producer. However, most of pulp, which is used to produce paper, is imported from abroad.

In an effort to meet the need for pulp materials, countries with poor forest resources like China, the Middle East and India are developing herbaceous agricultural wastes or bamboos into pulp materials. Even the sugarcane dregs discarded after sugar making are developed into pulp materials.

A new pulp material should be developed because fostering pulp industry results in destruction of forest resources. To do so, cellulose present in a variety of plants should be processed and treated to improve their value as paper materials. Rich countries in forest resources separate fibrous cellulose from wood to produce paper pulp and dissolving pulp. Therefore, over 90 % of pulp produced across the world is made from wood. However, with a plant distribution inappropriate for pulping and with a dearth of forest resources, Korea has to find a new strategy.

Corns cultivated in farms are used as food. But, cornstalks are mostly discarded even without being used as fodder. In the light of resources utilization and farmers' income augmentation, cornstalks need to be processed and treated to be useful for pulp or other materials, as in development of the Korean paper.

Every year, 750 million tons of cornstalks are produced worldwide. In the U.S.A alone, some 150 million tons of cornstalks are produced each year. But, cornstalks are not used in pulping and paper-making industries, as yet.

[8] Pulps can be classified into mechanical pulp, semichemical pulp and chemical pulp, depending on the pulping process. Mechanical pulp is manufactured by dissolving wood by mechanical grinding in the presence of water. Acicular trees having fiber le ngth, such as spruce, fir, pine and black pine, are pulped by this method. Semichemical

pulp is manufactured by steaming wood with a neutral sulfite solution and dissolving it through mechanical treatment. Light and soft trees, such as poplar, willow, linden tree, beech, oak, alder tree and ash tree, are pulped by this method. Chemical pulp is manufactured by adding a mixture solution of sulfurous acid and an acidic sulfite to a fibrous material and steaming it. Trees with low resin content are manufacture into chemical pulp for easy chemical treatment.

- [9] Referring to prior researches related with cornstalk, U.S. Patent No. 1,639,152, which was patented in the situation where quantitative experimentation was unavailable, disclosed a pulping process which comprised separating fibroid material from cornstalk by microbial fermentation and extracting pulp by using soda, lime and sulfite for use as lumber substitutes, wall boards, insulating materials, and so forth. Although the patent simply teaches that cornstalk can be steamed and dissolved by such chemicals as soda, lime, sulfite, and so forth to make paper for newspapers, it does not mention anything about specific composition of the cornstalk steaming solution, amount of addition thereof, steaming temperature or steaming time.
- [10] U.S. Patent No. 1,845,487 disclosed a method of preparing cellulose by steaming and dissolving plants with a small lignin content and a high pentosan (a pentose) content with dilute sulfuric acid or with sulfuric acid and a pressure of 10 pounds, and a pulping process of heating and pressing chipped or powdered cornstalks with a 1 % sulfuric acid solution to remove water-soluble materials.
- U.S. Patent No. 5,944,953 disclosed a pulping process for simultaneous mechanical and chemical defibration of cornstalks and straws. It mentions using soda (NaOH), lime soda (CaO<sub>2</sub>, NaOH) and neutral sulfite (Na<sub>2</sub>SO<sub>2</sub>, NaOH). Although not specifying the addition amount, it describes pulp making by refining cornstalks with 10-15 wt% of KOH and 1-5 wt% of K<sub>2</sub>SO<sub>3</sub>, based on the dry weight of the cornstalk, at 90 °C for 30-60 minutes.
- [12] Russian Patent No. 213995 describes a general herbaceous pulping process. This patent mentions nothing about solution composition, temperature or time of the steaming at the dissolving process.

#### Disclosure of Invention

#### **Technical Solution**

[13]

- It is an object of the present invention to provide a manufacturing method of semichemical mechanical pulp from cornstalks capable of offering environment-friendly pulp with less use of chemicals, offering a method of manufacturing specific Korean pulp using cornstalks and enabling manufacture of characteristic paper.
- [15] Hereunder is given a detailed description of the present invention.

The manufacturing method of semichemical mechanical pulp from cornstalks according to the present invention comprises: (1) a pretreatment process of cutting cornstalks to a size of 1 cm × 1 cm to 5 cm × 5 cm; (2) a chemical treatment process of adding a mixture of 0.05-3.0 wt% of caustic soda and 0.2-5 wt% of sodium sulfite, based on the total weight of the pretreated cornstalks, and applying heat and pressure in the range of 25-45 °C and 3-10 kg/cm²; (3) a first refining process of defibrating the chemical-treated cornstalks with a refiner; and (4) a second refining process of adding 5-20 wt% of hydrogen peroxide and 7-30 wt% of sodium silicate, based on the total weight of the pretreated cornstalks, to refine and bleach them.

[17] Hereinafter, the present invention is described further in detail referring to the preferred embodiments.

The manufacturing method of semichemical mechanical pulp from cornstalks according to the present invention is characterized by comprising: (1) a pretreatment process of cutting cornstalks to a size of 1 cm × 1 cm to 5 cm × 5 cm; (2) a chemical treatment process of adding a mixture of 0.05-3.0 wt% of caustic soda and 0.2-5 wt% of sodium sulfite, based on the total weight of the pretreated cornstalks, and applying heat and pressure in the range of 25-45 °C and 3-10 kg/cm²; (3) a first refining process of defibrating the chemical-treated cornstalks with a refiner; and (4) a second refining process of adding 5-20 wt% of hydrogen peroxide and 7-30 wt% of sodium silicate, based on the total weight of the pretreated cornstalks, while bleaching them.

[19] [20]

In (1) the pretreatment process, cornstalks are cut to a size of 1 cm  $\times$  1 cm to 5 cm  $\times$  5 cm for the succeeding chemical treatment process and refining process. Typically, a crusher or a grinder can be used for the purpose. On the average, the cornstalk is about 6 feet long and has leaves at about 2 feet or higher from the bottom. Thus, to about 3 feet from the bottom is adequate for pulping, but the present invention is not restricted thereto. Only considering that portion of the cornstalk, about 45-52.5 million tons of cornstalks adequate for pulping are produced each year in the U.S. alone. Worldwide, annual production of cornstalks adequate for pulping is estimated at 225-262.5 million tons. In the cornstalk pretreatment process, the size of the cornstalks to be pretreated is preferably in the range of 1 cm  $\times$  1 cm to 5 cm  $\times$  5 cm, although not particularly important. If the size of the cornstalks is below 1 cm  $\times$  1 cm, energy consumption increases. Otherwise, if it exceeds 5 cm  $\times$  5 cm, the succeeding chemical treatment process takes more time.

In (2) the chemical treatment process, a mixture solution of 0.05-3.0 wt% of caustic soda and 0.2-5 wt% of sodium sulfite, based on the total weight of the cornstalks, is added to the pretreated cornstalks and heat and pressure is applied in the range of 25-45 °C and 3-10 kg/cm<sup>2</sup>. Through this process, semichemical mechanical pulp is

obtained. If the addition amount of caustic soda is below 0.05 wt%, based on the total weight of the cornstalks, extraction of fibers may be difficult. Otherwise, if it exceeds 3.0 wt%, amount of fibers may decrease. If the addition amount of sodium sulfite is below 0.2 wt%, based on the total weight of the cornstalks, flexibility of fibers may decrease. Otherwise, if it exceeds 5 wt%, amount of fibers may decrease. Application of pressure may be performed with a screw press, etc.

- In (3) the first refining process, the chemical-treated cornstalks are defribrated with a refiner. In this process, such materials as resin are extracted and removed from the cornstalks and lignin is softened to improve fiber length and mechanical properties of the resultant pulp.
- In (4) the second refining process, 5-20 wt% of hydrogen peroxide and 7-30 wt% of sodium silicate, based on the total weight of the pretreated cornstalks, are added to the cornstalks to refine and bleach them. In this process, mechanical properties are further improved and made uniform. Also, high-quality pulp is obtained by bleaching. If the addition amount of hydrogen peroxide is below 5 wt%, bleaching may be insufficient. Otherwise, if it exceeds 20 wt%, fibers may be damaged. If the addition amount of sodium silicate is below 7 wt%, based on the total weight of the pretreated cornstalks, decomposition of hydrogen peroxide may be insufficient. Otherwise, if it exceeds 30 wt%, effect of excessive addition is minimal.
- After (1) the pretreatment process, a heat treatment process of heating the cornstalks to 50-80 °C may be included. Heat treatment in this temperature range may improve pulp extraction efficiency while reducing the amount of chemicals to be used. If the heat treatment is performed below 50 °C, mechanical pulping may be insufficient. Otherwise, if it is performed at higher than 80 °C, it is uneconomical. The heat treatment may be performed inside a conventional silo.

[25]

# **Best Mode for Carrying Out the Invention**

[26] Hereinafter, the present invention is described further in detail through examples. However, the following examples are only for the understanding of the present invention and they should not be construed as limiting the invention.

[27]

- [28] [Examples]
- [29] <u>Example 1</u>
- 1 kg of carefully selected cornstalks were crushed into chips of a size of 3 cm × 3 cm using a crusher. The resultant chips were put in a screw presser. 10 g of caustic soda (0.1 wt% based on the weight of the cornstalk chips) was added and refining was performed at 35 °C and normal pressure to obtain semichemical mechanical pulp.

Physical properties of the semichemical mechanical pulp were measured. It had a Freeness(CSF) of 85-1300, a tensile strength of 1.5-3.0 kgf, a breaking length of 2.0-3.0 km and a tear strength of 17-20 g. Thus, it was confirmed to be adequate for manufacturing printing paper or other paper.

[32] [33]

[34]

#### Example 2

- Semichemical mechanical pulp was manufactured in the same manner of Example 1, except that 140 g of caustic soda was used and a pressure of 7 kg/cm<sup>2</sup> was applied.
- Physical properties of the semichemical mechanical pulp were measured. It had a CSF of 85-1200, a tensile strength of 1.0-3.0 kgf, a breaking length of 2.0-3.0 km and a tear strength of 17-20 g. Thus, it was confirmed to be adequate for manufacturing printing paper or other paper.

[36] [37]

### Example 3

- 1 kg of carefully selected cornstalks were crushed into chips of a size of 3 cm × 3 cm using a crusher. The resultant chips were put in a screw press. 60 g of sodium sulfite (0.6 wt% based on the weight of the cornstalk chips) was added and refining was performed at 35 °C and normal pressure to obtain semichemical mechanical pulp.
- [39] Physical properties of the semichemical mechanical pulp were measured. It had a CSF of 85-1300, a tensile strength of 1.5-3.0 kgf, a breaking length of 2.0-3.0 km and a tear strength of 17-20 g. Thus, it was confirmed to be adequate for manufacturing printing paper or other paper.

[40]

#### [41] <u>Example 4</u>

- [42] Semichemical mechanical pulp was manufactured in the same manner of Example 3, except that 90 g of sodium sulfite was used and a pressure of 7 kg/cm<sup>2</sup> was applied.
- Physical properties of the semichemical mechanical pulp were measured. It had a CSF of 85-1200, a tensile strength of 1.5-3.0 kgf, a breaking length of 2.0-3.0 km and a tear strength of 17-20 g. Thus, it was confirmed to be adequate for manufacturing printing paper or other paper.

[44]

#### [45] Example 5

- 1 kg of carefully selected cornstalks were crushed into chips of a size of 3 cm × 3 cm using a crusher. The resultant chips were put in a screw press. 60 g of sodium bisulfite (0.6 wt% based on the weight of the cornstalk chips) was added and refining was performed at 35 °C and normal pressure to obtain semichemical mechanical pulp.
- Physical properties of the semichemical mechanical pulp were measured. It had a CSF of 85-1300, a tensile strength of 1.5-3.0 kgf, a breaking length of 2.0-3.0 km and a

tear strength of 17-20 g. Thus, it was confirmed to be adequate for manufacturing printing paper or other paper.

[48] [49]

#### Example 6

[50] Semichemical mechanical pulp was manufactured in the same manner of Example 5, except that 90 g of sodium bisulfite was used.

Physical properties of the semichemical mechanical pulp were measured. It had a CSF of 85-1200, a tensile strength of 1.5-3.0 kgf, a breaking length of 2.0-3.0 km and a tear strength of 17-20 g. Thus, it was confirmed to be adequate for manufacturing printing paper or other paper.

[52] [53]

#### Example 7

1 kg of carefully selected cornstalks were crushed into chips of a size of 3 cm × 3 cm using a crusher. The resultant chips were put in a screw press. 20 g of caustic soda and 30 g of sodium sulfite were added and refining was performed at 35 °C. Then, 15 g of hydrogen peroxide and 200 g of sodium silicate were added for refining and bleaching to obtain semichemical mechanical pulp.

[55] Physical properties of the semichemical mechanical pulp were measured. It had a CSF of 95-1050, a tensile strength of 2-4 kgf, a breaking length of 2.5-3.0 km, a tear strength of 19-25 g, a whiteness of 55-72 % and an opacity of 94-98. Thus, it was confirmed to be adequate for manufacturing printing paper or other paper.

[56] [57]

### Example 8

[58] Semichemical mechanical pulp was manufactured in the same manner of Example 7, except that the cornstalk chips were put in a silo and pretreatment was performed at 60 °C.

[59] Physical properties of the semichemical mechanical pulp were measured. It had a CSF of 95-1000, a tensile strength of 2-3 kgf, a breaking length of 2.5-3.0 km, a tear strength of 19-20 g, a whiteness of 61-67 % and an opacity of 96-98. Thus, it was confirmed to be adequate for manufacturing printing paper or other paper.

[60]

# **Industrial Applicability**

As apparent from above description, the present invention offers a process for manufacturing of the semichemical pulp of cornstalks, which are available almost infinitely without destroying forests but have not been used for pulping, into semichemical mechanical pulp using conventional mechanical pulping facilities with no special difficulties. Considering that vast forests had to be destroyed worldwide to produce paper,

the present invention offers the advantage of manufacturing semichemical pulp from cornstalks while protecting the forests.

[62] While the present invention has been described in detail with reference to the preferred embodiments, those skilled in the art will appreciate that various modifications and substitutions can be made thereto without departing from the spirit and scope of the present invention as set forth in the appended claims.

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# **Claims**

- [1] A method for manufacturing semichemical pulp from cornstalks comprising:

  (1) a pretreatment process of cutting cornstalks to a size of 1 cm × 1 cm to 5 cm × 5 cm; and
  - (2) a refining process of refining the pretreated cornstalks with 0.05-15 wt% of caustic soda (a steaming solution), based on the total weight of the cornstalks, at 25-85 °C and at normal pressure or a pressure of 3-10 kg/cm<sup>2</sup>.
- [2] A method for manufacturing semichemical pulp from cornstalks comprising:

  (1) a pretreatment process of cutting cornstalks to a size of 1 cm × 1 cm to 5 cm × 5 cm; and
  - (2) a refining process of refining the pretreated cornstalks with 0.5-10 wt% of sodium sulfite or sodium bisulfite (a steaming solution), based on the total weight of the cornstalks, at 25-85  $^{\circ}$ C and at normal pressure or a pressure of 3-10 kg/cm<sup>2</sup>.
- A method for manufacturing semichemical pulp from cornstalks comprising:

  (1) a pretreatment process of cutting cornstalks to a size of 1 cm × 1 cm to 5 cm × 5 cm;
  - (2) a chemical treatment process of adding a mixture solution of 0.05-3.0 wt% of caustic soda and 0.2-5 wt% of sodium sulfite, based on the total weight of the cornstalks, to the pretreated cornstalks and applying heat and pressure in the range of 25-45 °C and 3-10 kg/cm<sup>2</sup>;
  - (3) a first refining process of defibrating the chemical-treated cornstalks with a refiner; and
  - (4) a second refining process of adding 5-20 wt% of hydrogen peroxide and 7-30 wt% of sodium silicate, based on the total weight of the cornstalks, to the cornstalks to refine and bleach them.
- [4] The method of claim 1 further comprising a heat treatment process of heating the cornstalks to 50-80 °C after (1) the pretreatment process.

International application No. PCT/KR2005/001389

#### A. CLASSIFICATION OF SUBJECT MATTER

# IPC7 D21H 11/12

According to International Patent Classification (IPC) or to both national classification and IPC

#### B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols) IPC7 D21B 1/02, D21C 3/00, D21H 11/12

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched Korean Patents and applications for inventions since 1975

Electronic data base consulted during the intertnational search (name of data base and, where practicable, search terms used) eKIPASS

#### C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	KR 1992-0000746 B1 (JEONJU PAPER MFG. CO.) 21 Jan. 1992 see the claim	3
Y X	KR 10-324487 B1 (POLYCAM CO., LTD.) 27 Feb. 2002 see the claim	3 1, 2, 4
Α	JP 04-057982 A (HATA KOSEI) 25 Feb. 1992 see the whole document	1 - 4
Α .	US 5944953 A (Lavoie et al.) 31 Aug. 1999 see the whole document	1 - 4

- 1	
- 1	 Further documents are listed in the continuation of Box C.
	 Fullier documents are listed in the continuation of Box C.

See patent family annex.

- \* Special categories of cited documents:
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# INTERNATIONAL SEARCH REPORT

Information on patent family members

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Patent document cited in search report	Publication date	Patent family member(s)	Publication date
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KR 10-324487 B1	27.02.2002	None	
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